

mass spectrometer 10. Other numbers and shapes of the openings can be used. Permeation may be studied for the area of one opening position-resolved

Figure 7 shows the arrangement of the gas container 49 with the sample 50 and the lid 51 with respect to the enclosure 52 housing the mass spectrometer. If a hole of the lid 51 are in registry with the aperture 53 of the enclosure 52 of the mass spectrometer 10 gas or vapour molecules from the region of the sample that is located there will preferentially contribute to the partial pressure such creating a contrast picture of the rate of permeation through the sample. The gas container is located on the xyz-stage 9 and can be moved relative to the aperture 53 of the enclosure 52 of the mass spectrometer to expose different areas of the sample to the mass spectrometer. After finishing the measurement at one location, the sample can be shifted relative to the mass spectrometer for the mass spectrometer to face the next location. This way, the rate of permeation can be measured along a line or a two-dimensional pattern.

Figure 8 shows the principle of increasing the sensitivity by pumping the interior of the enclosure 52 housing the mass spectrometer 10 through a bypass 54 connected to the ion pump 14.

Figure 9 shows an example for a position-resolved measurement in an arrangement similar to figure 8. A polymer sample made of PEN 55 with an exposed area of about 1mm diameter was mounted on a gas container 56 containing water vapour. Outside the exposed sample area the sample was covered with an impermeable material 57. The gas container 56 was positioned on a xyz-stage 9 closely to the aperture 58 of the enclosure 59 of the mass spectrometer 10. It was then moved across the x-direction in steps of about 0.25 mm. At each location a few data points were taken. Figure 9 shows the measured partial pressure signal as function of lateral displacement of the sample. At $x=0$ the sample is in registry with the entrance aperture 58, which leads to a maximum in the partial pressure signal. At $x=0.5$ mm the exposed sample area of the sample has passed the projection of the entrance aperture 58 completely. The change in the partial pressure signal between $x=0$ and $x=0.5$ mm indicates areas of higher and lower permeability.

CLAIMS

What is claimed is:

1. A method of measuring the rate of permeation of gases or vapours or mixtures thereof through a test sample comprising:

- Providing an amount of gas or vapour in a gas container at a certain vapour or gas pressure;
- Arranging the container containing the gas or vapour in a vacuum chamber, which is under vacuum such that the gas or vapour permeating from the container through the test sample communicates with the vacuum chamber under vacuum
- Providing a means to change the relative position between the gas container with the test sample and the mass spectrometer for position resolved measurement of permeation

- Using a mass spectrometer to detect the partial pressure of the gas or vapour after permeation through the test sample
- Estimating the rate of permeation from the signal measured by the mass spectrometer

2. The method as claimed in claim 1 wherein the gas container comprises a body, to which one or more test samples are attached forming a seal.
3. The method as claimed in claim 2 wherein one or more of the remaining openings of the body of the gas container are used to accommodate a closing device such as a valve to separate the interior of the gas container from its surrounding the outside.
4. The method as claimed in claim 1 where the pressure decrease inside the gas container due to permeation is compensated by a flexible device that can change its inner volume.
5. The method as claimed in claim 2 wherein the test sample is sealed to the gas container by pressing the test sample against a sealing face of the gas container with or without using a gasket
6. The method as claimed in claim 2 wherein the test sample is sealed to the gas container by an adhesive
7. The method as claimed in claim 2 wherein the test sample is a film
8. The method as claimed in claim 1 where the gas container is filled with gas or vapour inside a vacuum chamber
9. The method as claimed in claim 8 where the gas container is filled through a hole with a closing device communicating with a filling chamber filled with a gas or a vapour.
10. The method as claimed in claim 9 where the closing device is operated by means of a tool on a feedthrough.
11. The method as claimed in claim 2 where the temperature of the gas container with the test sample is changed by cooling or heating over a wide temperature range, including cryogenic temperatures.
12. The method as claimed in claim 2 wherein the test sample is made of a polymer, a metal, a ceramic, a biological material or a combination thereof.
13. The method as claimed in claim 1 where the partial pressure is measured with the mass spectrometer after the signal has stabilised to a constant value.

14. The method according to claim 1 where the rate of permeation of a gas or vapour is determined from the partial pressure (measured by a mass spectrometer) and calibration against the rate of permeation of gas or vapour of reference samples.
15. The method according to claim 14 where the partial pressure measured from the reference samples is used for calibration
16. The method according to claim 1 where the experiment is carried out in a high vacuum (HV), ultra-high vacuum (UHV) chamber or extra-high vacuum (XHV) chamber under vacuum.
17. The method according to claim 1 where the vapour is water vapour.
18. A method of measuring the rate of permeation of water vapour through a test sample comprising:
- providing an amount of water vapour in a container at a certain relative humidity;
 - arranging the container containing the water vapour in a vacuum chamber under vacuum such that the water vapour permeating through the test sample communicates with the vacuum chamber under vacuum
 - using a mass spectrometer to detect the partial pressure of the water vapour after permeation of the water vapour through the test sample;
 - providing a means to change the relative position between gas container with test sample and the mass spectrometer
 - deriving the rate of water vapour permeation from the signal measured by the mass spectrometer
19. The method as claimed in claim 18 wherein the gas container comprises a body, to which a test sample is attached forming a seal covering an opening of the body of the gas container.
20. The method as claimed in claim 19 wherein one or more of the remaining openings of the gas container are used to house a closing device such as a valve to separate the interior of the gas container from its surrounding.
21. The method as claimed in claim 19 wherein the test sample is a film
22. The method as claimed in claim 18 where the gas container has a movable part for changing the internal volume.
23. The method as claimed in claim 18 where the gas container is filled with water vapour inside a vacuum chamber.
24. The method as claimed in claim 18 where the gas container is filled through a hole with a closing device inside a vacuum chamber with water vapour.

25. The method as claimed in claim 18 where the gas container containing a reservoir with water vapour is filled with vapour from this reservoir.
26. The method as claimed in claim 18 where the water vapour pressure can be changed.
27. The method as claimed in claim 18 where the temperature of the gas container with the test sample is changed by cooling or heating
28. The method as claimed in claim 19 wherein the test sample is made of a polymer, a metal, a ceramic, a biological material or a combination thereof
29. The method as claimed in claim 18 where the partial pressure is measured with the mass spectrometer after the signal has stabilised to a constant value.
30. The method according to claim 18 where the rate of permeation of water vapour is determined from the partial pressure as measured by a mass spectrometer and calibration against the rate of permeation of water vapour of reference samples.
31. The method according to claim 18 where the experiment is carried out in an ultra-high vacuum (UHV) chamber under vacuum or in a extra-high vacuum (XHV) chamber under vacuum or in a high vacuum (HV) chamber under vacuum.
32. The method according to claim 19 where the effective permeation area of the test sample is a few mm or less in one or two dimensions.
33. The method as claimed in claim 19 where the gas container contains a salt solution, which produces a characteristic relative humidity inside the gas container.
34. The method as claimed in claim 18 where the signal to noise ratio of the signal detected by the mass spectrometer is reduced by choosing a suitable isotope from $D_2^{16}O$, $D_2^{17}O$, $D_2^{18}O$, $H_2^{16}O$, $H_2^{17}O$, and $H_2^{18}O$.
35. The method according to claim 18 where the rate of water vapour permeation is determined from the partial pressure as measured by a mass spectrometer and calibration against the partial pressure of one or more samples with a known rate of water vapour permeation.
36. A method where the rate of permeation is estimated on test samples other than a film, including batteries and complete microelectronic assemblies.
37. The method according to claim 36 where the test sample is filled with a gas or a vapour or a mixture thereof through a filling facility such as a valve.

38. The method according to claim 36 where the test sample is filled with a gas or a vapour or a mixture thereof inside a filling chamber.
39. The method according to claim 36 where the test sample is a resin between two parts of the gas container.
40. The method according to claim 36 where the rate of permeation through an edge of a sample is measured.
41. The method according to claim 36 where the gas container has a movable part for changing the internal volume of the gas container.
42. A method where the rate of permeation is studied position-resolved on different locations on the test sample.
43. The method according to claim 42 where the test sample and the mass spectrometer are movable with respect to each other.
44. The method according to claim 42 where a stage holding the sample is used to move the sample with respect to the fixed mass spectrometer
45. The method according to claim 42 where the mass spectrometer is covered in an enclosure containing at least one hole through which the gas enters the mass spectrometer after permeation through the test sample
46. The method according to claim 42 where the enclosure has a conical bottom with a hole and a tube attached to the hole for position-resolved measurements
47. The method according to claim 42 where the gas container has a lid with parallel face and holes extending through the whole thickness of the lid.
48. The method according to claim 42 where the enclosure housing the mass spectrometer is evacuated by a separate pump or through a bypass to the existing pump on the vacuum chamber.
49. The method according to claim 42 where the gas container has a movable part for changing the internal volume of the gas container.
50. An apparatus for measuring the rate of permeation of a gas or vapour (including water vapour) consisting of:
- one or more vacuum chambers
 - a gas container which is removable from the vacuum system
 - a filling facility
 - a mass spectrometer for partial pressure measurement
 - a means for changing the relative positions of mass spectrometer and test sample

- a means of transferring the gas container with the test sample.

51. The apparatus according to claim 50 having a gas container that can be filled by means of a filling facility provided in one of the vacuum chambers.

52. The apparatus according to claim 50 with the investigation chamber is under HV, UHV or XHV conditions.

53. The apparatus according to claim 50 where the gas container can be moved between vacuum chambers and removed from the vacuum chambers.

54. The apparatus according to claim 50 where the size of test sample is less than a mm to a few mm in one or two dimensions.

55. The apparatus according to claim 50 where the test sample is clamped or glued to the gas container

56. The apparatus according to claim 50 where the test sample is a film or a device or an assembly of several parts of a device.

57. The apparatus according to claim 50 where the gas container contains gas or vapour including water vapour.

58. The apparatus according to claim 50 where the pressure of the gas or vapour inside the gas container can be varied by means of a movable part for changing the internal volume of the gas container.

59. The apparatus as claimed in claim 50 where the partial pressure is measured with the mass spectrometer after the signal has stabilised to a constant value.

60. The apparatus according to claim 50 where the rate of permeation is estimated from the measured partial pressure and a calibration against one or more samples with a known rate of permeation.

61. The apparatus according to claim 50 where the temperature of the test sample can be varied.

62. The apparatus according to claim 50 where the mass spectrometer has an enclosure, which can be pumped.